

**EPITAXIAL LATERAL OVERGROWTH OF II-VI
SEMICONDUCTORS**

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By

Prof. Ishwara Bhat

JEC 6003, ECSE Department

Rensselaer Polytechnic Institute

Troy, NY 12180-3590

Ph: 518-276-2786; Fax: 518-276-2433

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14. ABSTRACT Epitaxial lateral overgrowth of CdTe was carried out on Si and GaAs substrates. Perfectly selective epitaxial growth was demonstrated on CdTe/Si and GaAs substrates using Si ₃ N ₄ as the mask material. Selective growth has been possible at T>500°C and pressure <30 Torr. Lateral to vertical growth rate ratio of >3 has been achieved. Reduction in defect density in films has been demonstrated.					
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Appendix

1. Journal article titled "Epitaxial Lateral Overgrowth of CdTe on Si and GaAs Substrates Using Metal organic Vapor Phase Epitaxy"
2. Journal article titled "Atomic Force Microscopy Study of CdTe Films Grown by Epitaxial Lateral Overgrowth".

Abstract

Under this contract, epitaxial lateral overgrowth of CdTe was carried out using MOCVD. The growth was carried out on Si, GaAs and CdTe/Si substrates, and the efficacy of ELO method to reduce the dislocation density in heteroepitaxial layers were assessed. More than 100 growth runs were carried out using a home-built vertical low-pressure MOCVD reactor operated at low pressure. This report describes the summary of the work and attached manuscripts describe the detailed results. Several conference presentations (over 5) and two manuscripts were submitted with one more in preparation.

The results of the work shows that perfectly selective CdTe growth can be carried out on both Si and GaAs substrates using Si_3N_4 as the mask layer. At temperatures higher than 500°C , and pressures lower than about 30 Torr, Si_3N_4 mask layer is completely non-wettable, and growth takes place only on the window openings. Depending on the substrate orientation, the growth rates along different directions are drastically different, and hence the lateral to vertical growth rate ratio is different for different wafer orientations. Lateral to vertical growth rate ratio over 3 can be obtained under certain conditions. Substrates including GaAs(100), GaAs(110), GaAs(111)A, GaAs(111)B and CdTe(211)/Si(111) were used in this study. The crystal phases of the films were studied by single crystal and double crystal X-ray diffractions. The selectivity and the morphology of the films were studied by SEM and AFM.

AFM studies reveal that the diffusion lengths of the precursor atoms are large ($>50\mu\text{m}$) for the conditions used for CdTe selective growth. As a result, the crystal grain size can be up to $100\mu\text{m}$ in size. This property can be used to make two-dimensional IR micro-lenses in any arbitrary substrates. From AFM measurements, it appears that most of the layers are free of dislocations except at the grain boundary when the growth windows are properly aligned in certain crystallographic orientation. In order to further investigate the growth anisotropy, different substrates with stripes of growth window oriented in different directions have been studied. The results show that ELO growth can be an important growth method that can be used for the hetero-epitaxial growth of defect free layers on alternate substrates.

List of Publications

Following is the list of publications/presentations arising from this contract.

Conference Presentations

1. I. Bhat, "Epitaxial growth of CdTe on Silicon substrates for HgCdTe Epitaxy", Invited talk at the 11th American Conference on Crystal Growth and Epitaxy", August 1-6, 1999.
2. R. Zhang and I. Bhat, "Epitaxial lateral overgrowth of CdTe on Si and GaAs substrates", presented at the 1999 II-VI workshop, Las Vegas, NV, September 22-24, 1999.
3. I. Bhat and R. Zhang, "Characterization of CdTe grown on GaAs by lateral epitaxial overgrowth technique, presented at the 2000 II-VI workshop, Albuquerque, NM, October 30- November 1, 2000.
4. I. Bhat and R. Zhang, "Epitaxial lateral overgrowth of II-VI compounds: Growth mechanisms", 2000 Electronics Materials Conference, University of Denver, Colorado, June 21-23, 2000.
5. I. Bhat and R. Zhang, "Epitaxial lateral overgrowth of CdTe on GaAs using MOVPE", presented at the 10th biennial workshop on MOVPE, March 11-15, 2001, San Diego, CA.

Journal Publications

1. R. Zhang, and I. Bhat, "Selective growth of CdTe on Silicon and GaAs substrates", J. Electron. Mater., V29, p765-769, 2000.
2. R. Zhang and I. Bhat, "Atomic Force Microscopy Studies of CdTe Films Grown by Epitaxial Lateral Overgrowth", submitted to J. Electron. Mater., 2001.
3. I. Bhat and R. Zhang, "Anisotropy of Growth in Selective MOVPE of CdTe Films on GaAs and CdTe Substrates", in preparation.

1. Introduction

Infrared detector material, Mercury cadmium telluride ($\text{Hg}_{1-x}\text{Cd}_x\text{Te}$), is generally grown on CdTe substrates because its lattice constant at any x value is closely matched to that of CdTe. As a first step to grow low-defect-density HgCdTe, high quality CdTe and CdZnTe layers of suitable orientation have to be grown on Si substrates. Therefore, hetero-epitaxial growth of CdTe on Si has been actively studied for the development of large area substrates suitable for HgCdTe growth. Epitaxial Lateral Overgrowth (ELO) technique used for the growth of GaN on sapphire substrate has been reported to be an effective way to reduce the threading dislocations in the films. The objective of this research is to investigate the possibility of applying ELO technique to grow low-defect-density CdTe and to study the effects of growth conditions on the selective growth of CdTe.

Our previous studies demonstrate that perfect selectivity can be achieved for the growth of CdTe on patterned GaAs substrate using Si_3N_4 mask layer.^{1,2} Further systematic investigation of the effects of the growth conditions on the selectivity and the material properties have been carried out. The selectivity and crystal orientation of the films were assessed through scanning electron microscope, single crystal x-ray diffraction and double crystal rocking curves. The morphology and dislocation of the films were studied by SEM and AFM, and the results were compared with those of the CdTe films grown directly on unpatterned substrates. It is demonstrated that the threading dislocation density of the CdTe films can be reduced by the lateral overgrowth as well as the patterned growth. A possible nucleation and growth model on the patterned substrates were introduced based on our experimental results.

2. Work performed during the program period

2.1 Optimization of the growth conditions for ELO-CdTe growth

For the period from June 1998 to January 1999, we focused on setting up the CVD system. From January 1999 to July 1999, we carried out several growths to obtain selective CdTe on Si substrates. From July 1999 till June 2000, we have focused our efforts on optimizing the growth condition that is favored by the selective CdTe growth on different substrates. The depositions were carried out using the vertical MOCVD reactor that was previously used for HgCdTe epitaxy. Dimethylcadmium (DMCd) and Diethyltelluride (DETe) were used as Cd and Te sources. Highly purified H₂ gas was used as the carrier gas for precursors. Commercially available GaAs(100), GaAs(111)A, GaAs(111)B and CdTe(211)/Si(111) (supplied by Dr. Nibir Dhar, ARL) substrates were used. Based on our previous results for the selective epitaxy of CdTe, Si₃N₄ was used as the mask material. A thin Si₃N₄ layer of about 100 nm thick was first deposited by PECVD on all substrates and then patterned using standard photolithography process. The deposition of Si₃N₄ mask layer and the patterning of all the substrates were carried out in a class 100 clean. Mask#1 was specially designed for the study of the patterned growth. It contained groups of lines, with widths varying from 1 μm to 20 μm, and the spacing between the lines varying from 3 μm to 500 μm. The lines are oriented in four orthogonal directions. Before deposition, patterned substrates were cleaned by xylene at 200°C and then by diluted NH₄OH to remove thin native oxide layer.

A series of CdTe growth runs, about 50 runs, were carried out in order to systematically evaluate the growth conditions for CdTe selective growth on patterned GaAs and CdTe(211)/Si substrates. The morphology of the films was studied by Joel Model JSM-840 SEM, and the vertical and the lateral growth rates of the films were also measured by SEM. For most of the runs, the reactor pressure was kept constant at 25 torr and total gas flow was kept constant at 2L/min. The substrate temperatures were varied in the range of 475°C to 550°C, and the precursor flows were also varied systematically. Under some conditions, especially at pressures less than 20 Torr, CdTe did not grow on

the substrates and the substrates were etched off by the precursor atoms instead. The etching was confirmed by the Alfa-step measurements.

The experimental results show that growths of CdTe on patterned GaAs(100), GaAs(111)A, GaAs(111)B, and CdTe(211)/Si wafers can all achieve perfect selectivity. To obtain good selective growth, the temperature of the substrate needs to be not lower than 530°C for GaAs substrates. Using CdTe(211)/Si substrates, the patterned CdTe growth can maintain good selectivity at lower temperature by adjusting the precursor flows. The lowest substrate temperature that can result in good selectivity for CdTe(211)/Si substrates is 495°C. It was also found that the growths were anisotropic on all types of substrates. However, patterned CdTe growth with smooth surface and vertical side-walls was only obtained on GaAs(100) substrates so far under certain growth conditions. Figure 1 shows a SEM picture of the CdTe stripes with smooth surface. The crystalline phases of the CdTe films were measured by single crystal and double crystal X-ray diffraction, as shown in Figure 2. Detailed discussion of the growth anisotropy will be written up as a manuscript³.

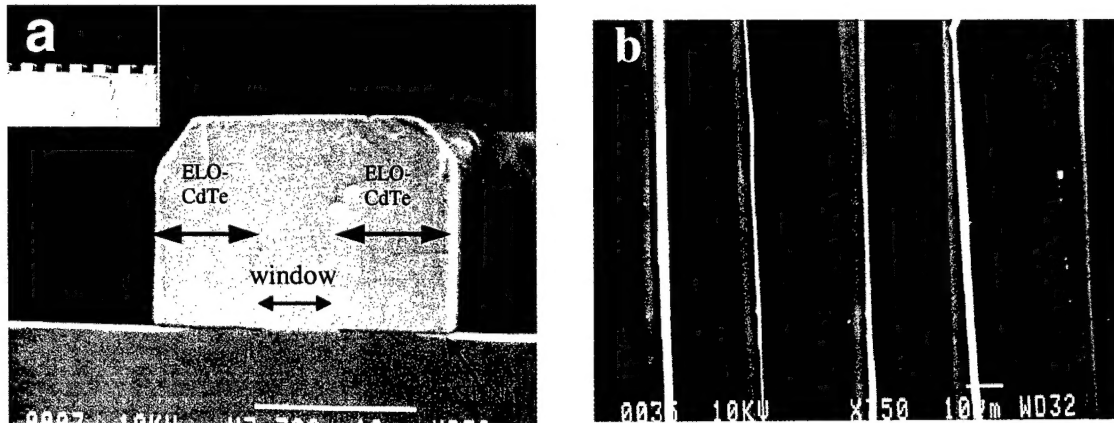


Figure 1. SEM pictures of CdTe grown on GaAs(100) substrate with stripe pattern along [011], $T_s=550^\circ\text{C}$, 5 μm growth window, (a) cross section of one stripe(the upper left corner is a picture of the cross section of stripes taken at lower magnification), (b) top surface of stripes. (scales in pictures are equal to 10 μm).

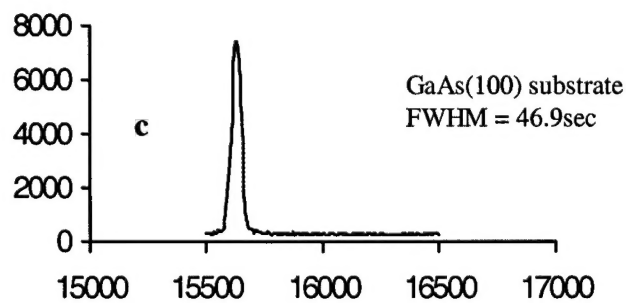
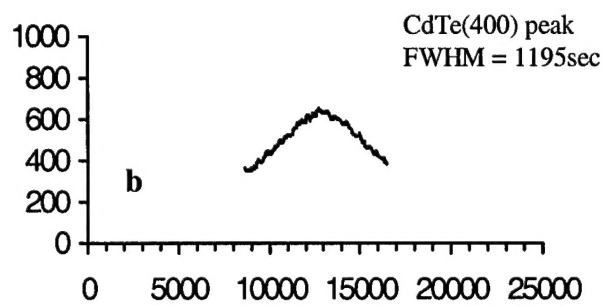
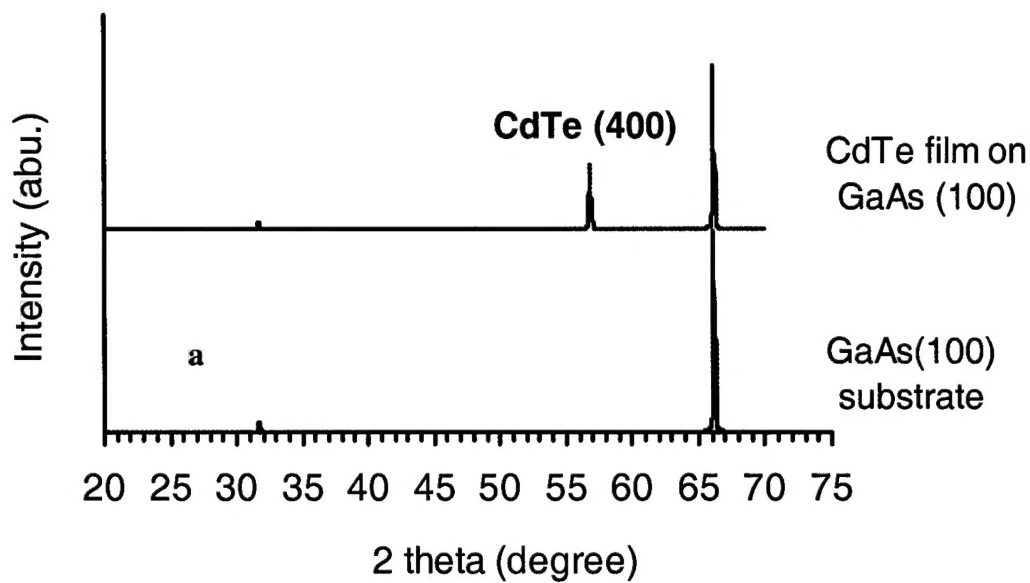


Figure 2. (a) Single crystal x-ray diffraction of CdTe stripes in figure 1 grown on GaAs(100) substrate. (b) Double crystal x-ray diffraction (DCD) of the stripes (c) DCD of GaAs substrate through the Si₃N₄ mask area. The FWHM of the CdTe peak is relatively large possibly caused by slightly different orientations of different stripes.

Lateral growth and surface diffusion are also studied. The lateral growth of the CdTe depends not only on the differences of the growth rates of the crystallographic planes, but also on the diffusion of the precursor atoms on the surface of substrate. The mask#1 we used consists of groups of stripes of various widths and various spaces. This can be used to study the surface diffusion lengths of precursor molecules during the growth, which is essential to determine the growth conditions necessary for ELO. Figure 3 shows the effect of the spacing between the growth windows on the lateral growth rate. The window spacing of 500 μm is considered as “infinite” spacing here and the lateral growth rate of these windows is considered as the saturated growth rate. A depletion zone model was used to study the surface diffusion and its influence on the lateral growth. The surface diffusion depletion zone, or the critical spacing, can be experimentally estimated taking the growth rate of the infinite spacing windows as the equilibrium value. Detailed discussion on the surface diffusion is included in the attached manuscript ^{1,3}.

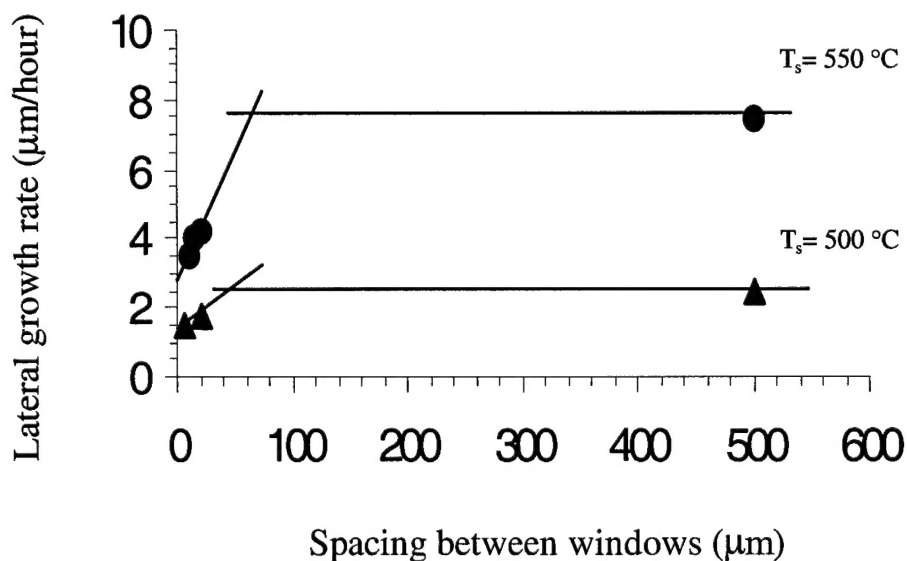


Figure 3. Dependence of the lateral growth of CdTe on the spacing of the growth windows. As the growth window spacing is reduced, the lateral growth rate reduces as expected.

2.2 Growth mechanism

The key advantage of the ELO technique is that the threading dislocations generated in the interface between the film and the substrate are not likely to propagate through the lateral overgrowth region. Numerous works have been done on the ELO of GaN, and it has been reported that the dislocation density of the ELO GaN can be reduced by 2 to 3 orders to 10^{-5} dislocations/cm² comparing to that of the conventional GaN.^{4,5} It has also been reported in the ELO studies of GaN films that the dislocations in the lateral overgrowth region is much less than that in the area above the growth window, or even no dislocation pit can be observed in the lateral overgrowth region by TEM or AFM.⁶⁻⁸ Similar results on the dislocation reduction are expected in our attempt to apply ELO to the growth of CdTe. Our results show that the dislocation density in the CdTe films grown by the selective epitaxy can indeed be reduced, however, might be by different mechanisms.

TEM is a common way to study the dislocations in thin films. However, our attempt of TEM study for the CdTe films is inconclusive because significant amount of ion milling damage was observed in CdTe. On the other hand, atomic force microscopy (AFM) turns out to be a powerful tool for the dislocation and morphology studies for the CdTe films.

When CdTe is grown on unpatterned wafer under the same conditions that is used for selective growth, we observed large facets. Usually the diffusion lengths of the precursor molecules are comparable to the size of the facet. Our results show that the grain size of CdTe grown on un-patterned GaAs (100) substrates can be as large as 100 μm . For a 100 μm facet, the diffusion length of the precursor molecule can be as large as 50 μm . In the selective CdTe growth with narrow windows (5 μm wide), facets also developed but the top surface is much more smoother. As we discussed in our attached manuscript "Atomic Force Microscopy Study of CdTe Films Grown by Epitaxial Lateral Overgrowth",⁹ the large diffusion length helps reduce the dislocation in the ELO CdTe films. Because the substrates are patterned before growth and the masked areas are un-wettable by CdTe, the nucleation of CdTe take place only in the growth window area. The walls of the Si₃N₄ mask layer inside the growth windows provide favorable nucleation sites. Figure 4 shows a

typical micrograph of ELO grown CdTe showing the presence of grain boundaries either perpendicular to the stripe direction or at an angle of 45 degrees.

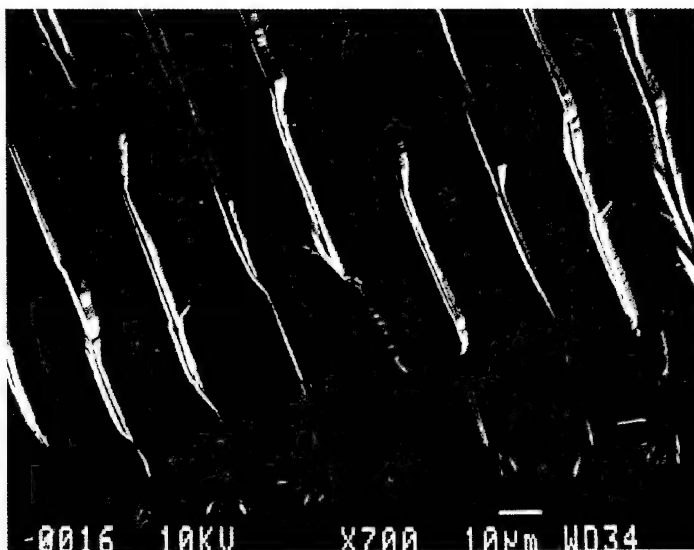
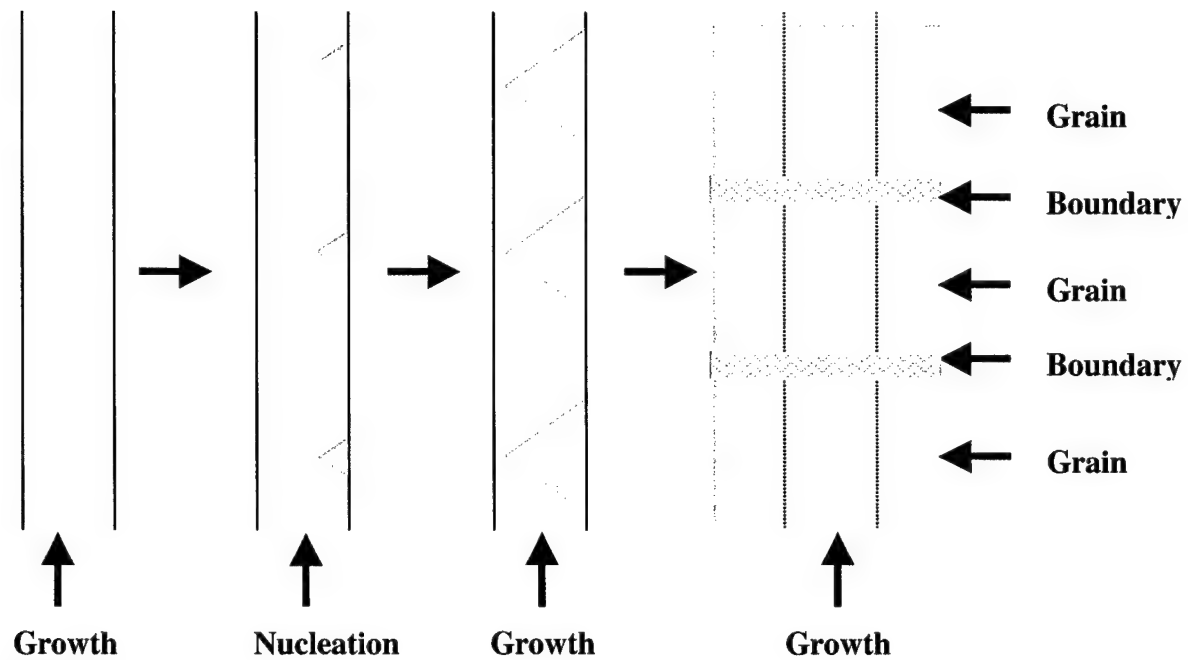
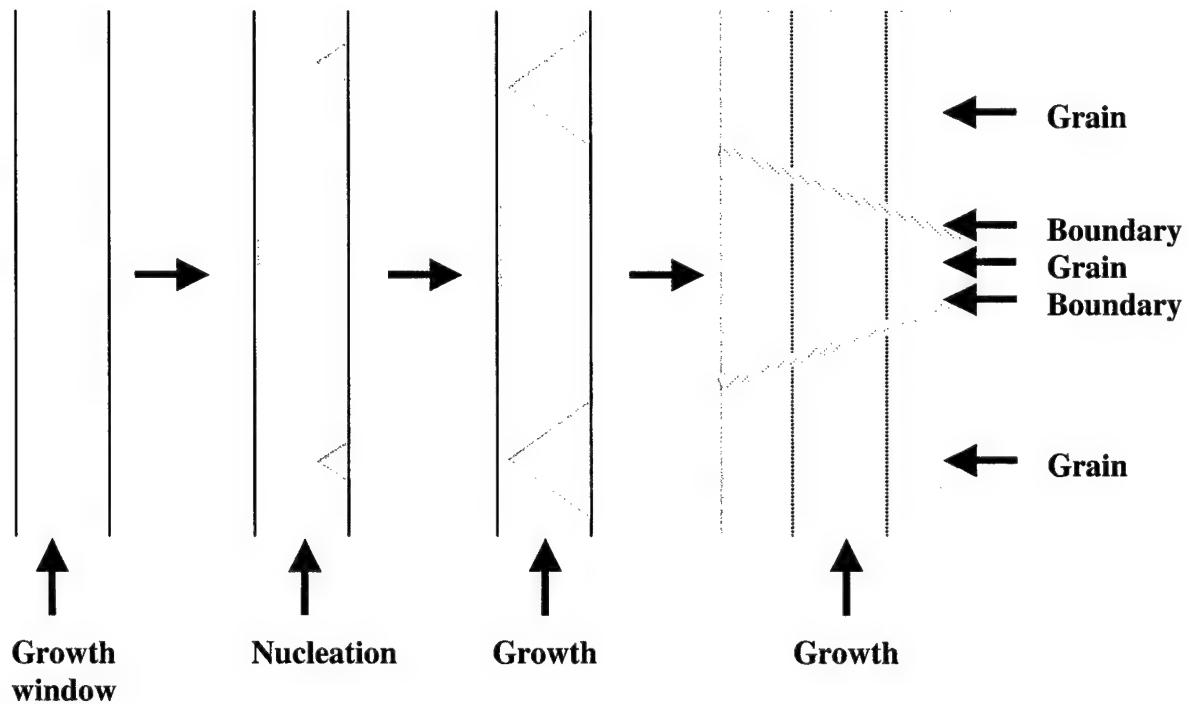


Figure 4 SEM micrograph of CdTe stripes with 5um windows. Note the presence of facets, and the facet edges are either perpendicular to the stripe directions or at an angle of 45 degrees.

Figure 5 schematically shows a possible nucleation and growth mechanism during the CdTe selective epitaxy. Figure 5(a) represents the case that two adjacent nucleus are in the same side of the window walls. When the two grains coalesce, the grain boundary is perpendicular to the stripe direction. The grain boundary has wavy step structure with high density of dislocation outlets when observed under AFM (shown in the attached manuscript). This type of grain boundary is referred to as type I. Figure 5(b) represents the case that two adjacent nucleus are in the opposite sides of the window walls. When the two grains coalesce, the grain boundary is at an angle to the stripe direction, and the atomic step structure near the grain looks more orderly. This type of grain boundary is referred as type II grain boundary. Between the grain boundaries, no dislocation outlets are seen.



(a). Type I grain boundary.



(b). Type II grain boundary.

Figure 5. Possible growth mechanism: schematic diagram of two types of grain boundaries in selective CdTe growth. (a). Type I grain boundary (b) Type II Grain Boundary.

AFM reveals that the CdTe grown in windows oriented along [110] contains only one crystal grain in the direction across the window due to the large diffusion length of precursor atoms. Nuclei line up along the window stripe and eventually grow into big grain in the size of several tens of microns. This makes it more important to orientate the growth window in proper direction. The dislocation density as determined by the dislocation outlets in AFM is negligible in both the window region and the ELO-grown region. The dislocation outlets are seen only along the grain boundary region.

The anisotropy of the CdTe growth on different types of substrates are studied. Figure 6 shows two SEM pictures of CdTe grown in the same run on windows oriented along different directions. The stripes along [110] have smooth surface and vertical sidewalls. The growth shown in Figure 6(b) might be due to the competing growth of several grains within the 5 μm growth window. As shown in the AFM section analyses in Figure 7, three grains can grow competitively within the 5 μm growth window. One possible reason for this is that the nucleation sites are defined by the steps on the GaAs (100) surface and also by the silicon nitride mask edge. These steps may arise due to the fact that the substrates are misoriented by a few degrees with respect to the (100) surface. Therefore, the number of surface steps available for nucleation across the 5 μm window depends on the window orientation. Different types of substrates, including GaAs(100), GaAs(111)A, GaAs(111)B, and CdTe(211)/Si have also been used to study the anisotropic growth of CdTe. Detail discussions are to be included in a manuscript titled "Anisotropy of Growth in Selective MOVPE of CdTe Films grown on GaAs and CdTe Substrates".³

Based on the above arguments, the dislocations in the CdTe films grown by selective epitaxy can be reduced not only by the lateral growth, but also by the patterned growth itself. In the conventional approach, CdTe film is grown on a planar wafer. In the selective growth, CdTe nucleation and growth are restricted only in the window region. The strain in the film can be greatly reduced because the lateral growth is free of confinement from other grains. Moreover, dislocation can also be reduced by the patterned growth due to the large diffusion length of precursor atoms and that the misfit dislocation can end at the Si_3N_4 mask edge. This is different from the growth of GaN because the nucleation density is much higher in the GaN growth.

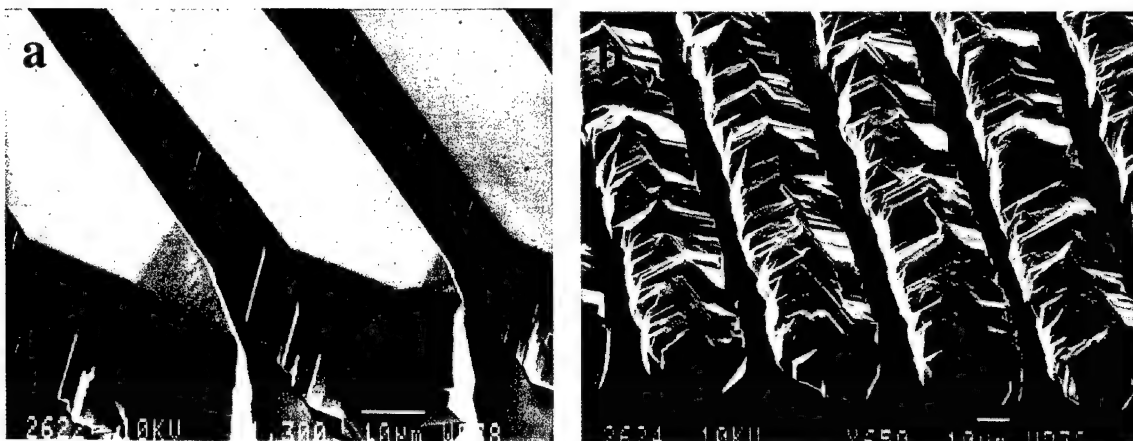


Figure 6. SEM- top surface of CdTe grown on GaAs(100) substrate. (a). stripe pattern along [110] (b) stripe pattern along [001].

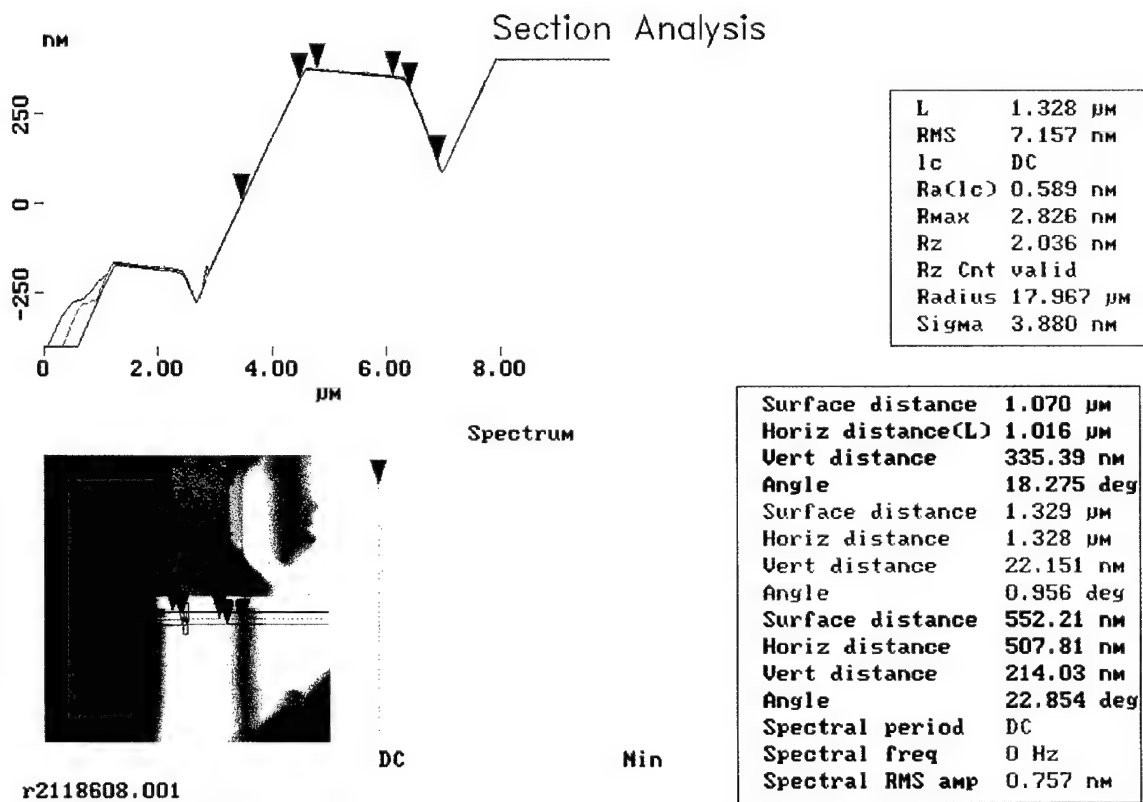


Figure 7. AFM analysis of the anisotropic CdTe growth on GaAs(100) substrate: Three grains grown competitively within the 5 μm growth window when the window orientation is not along (110) direction.

2.3 Lateral to vertical growth rate ratio

We have investigated the lateral and vertical growth rates for CdTe grown on GaAs(100) substrates. Figure 8 shows the ratio of the lateral to vertical growth rates (L/V ratio) as a function of the fill factor for CdTe growth. Fill factor is defined as the ratio of window width to the mask spacing, and zero fill factor corresponds to mask spacing of 100 μm or higher. Figure 8(a) compares the effect of the fill factor on the L/V ratio at different temperatures and (b) compares the effect of the fill factor on the L/V ratio for different flow rates of the precursors. Generally, the ratio L/V increases as the fill factor decreases. When the substrate temperature is increased from 530°C to 550°C, the L/V ratio slightly increases if the fill factor is larger than 0.2 or increases significantly if the fill factor is smaller than 0.2. The increase of the precursor flows has the same effect on the L/V ratio for all fill factors. The fact that increasing the precursor flow can increase the L/V ratio demonstrates that the CdTe growth under the conditions for selective epitaxy is mass transfer limited. In the CdTe selective epitaxy, the amount of the precursor delivered to the surface of the substrate in a certain period need to be kept small to prevent the nucleation on the mask area. Thus, the growth rates are limited by the amount of the precursor atoms that can reach the top and the side surfaces of the CdTe stripes. The L/V ratio can be increased by increasing the growth temperature because the diffusion of the precursor atoms is easier at higher temperature and this makes it possible for more atoms to reach the side surface of the stripes during the growth. The fact that the L/V ratio varies with the fill factor is also because the growth is controlled in the mass transfer limited region. The fill factor can affect the local reaction equilibrium during the deposition and thus change the L/V ratio. It is interesting to note that L/V ratio higher than 3 can be obtained.

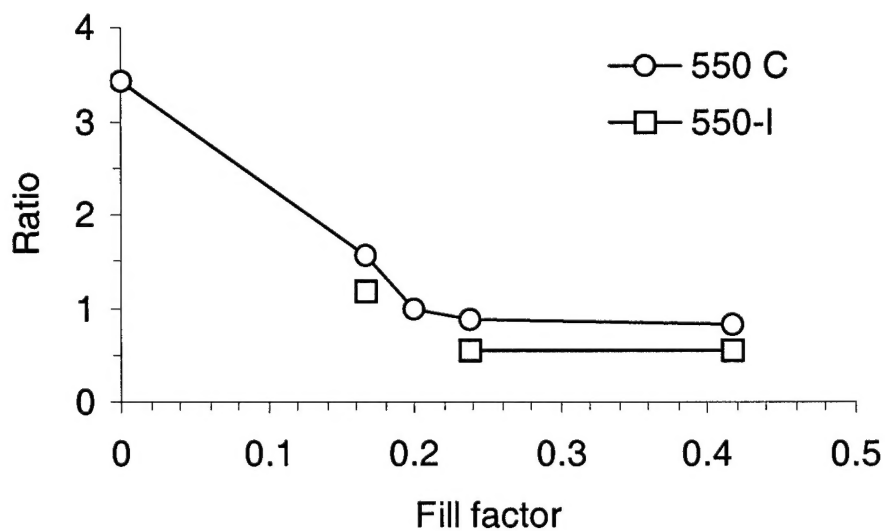
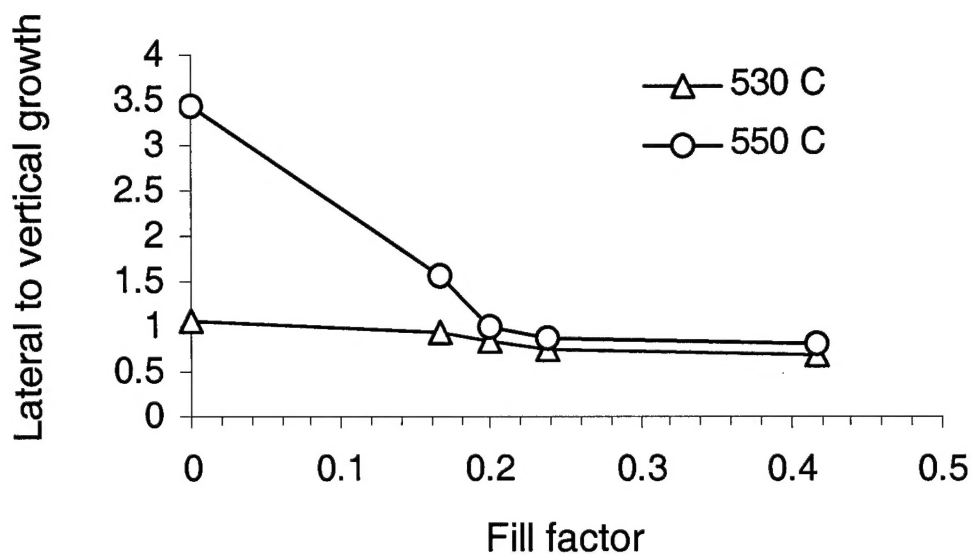


Figure 8. Ratio of lateral to vertical growth rate as a function of the fill factor of window pattern. (a) for two different growth temperatures. (b) for two different flow rates of precursors at the same growth temperature.

3. Conclusions

Under this contract, we successfully demonstrated that epitaxial lateral overgrowth is an attractive technique to grow defect-free CdTe on alternate substrates such as GaAs or Si. It appears that using two-dimensional window patterns, it is possible to grow defect-free CdTe grains of 100 μ m in sizes across over a large area. These large-grain CdTe can be used for either growing HgCdTe films or for use as two-dimensional CdTe templates. Complete coalescence of these grains into a full wafer form was not found to be achievable in our studies. Results are summarized as follows:

1. Selective epitaxy of CdTe can be achieved by using Si₃N₄ mask and controlling growth conditions. To achieve perfectly selective CdTe growth, the substrate temperature need to be higher than 495°C for CdTe(211)/Si substrates and higher than 530°C for GaAs substrates.
2. The CdTe growth rates are different on different substrates and are an isotropic along different crystallographic directions. CdTe can be obtained with smooth atomically flat surface and vertical side walls by suitably orienting window pattern directions and optimizing growth conditions.
3. The diffusion lengths of the precursor atoms are about 50-100 μ m at the growth conditions used for selective growth. The growth mechanism of CdTe is closely related to the high mobility of precursor atoms. Unlike GaN, the initial CdTe nuclei are far apart and these nuclei grow and merge to result in large grains.
4. From AFM measurements, it appears that most of the layers are free of dislocations, except at the grain boundaries. Evidence of dislocations is seen near the grain boundary where two grains meet. The dislocation reduction can be enhanced by controlling the growth window orientation and size. The growth window defines where the nucleation starts. Due to the large diffusion length, single crystal grains are lined up only along the stripe direction, several tens of microns apart. Thus, the

dislocation can be reduced not only by the lateral overgrowth, but also by the patterned growth approach itself. By using a two dimensional arrays of windows, it is possible to grow two-dimensional arrays of defect-free single crystal CdTe grains.

4.0 Acknowledgements

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